Journal of Advances in Biology & Biotechnology

Volume 27, Issue 7, Page 86-95, 2024; Article no.JABB.117231
ISSN: 2394-1081

Comparative Analysis of Nickel Adsorption by Natural Sisal Fiber and Treated with Citric Acid

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Authors' contributions

This work was carried out in collaboration among all authors. Author DGK participated in the conception, laboratory analyzes and final review. Authors AMdS and GBCJ contributed to the bibliographic review, data analysis and textual review. All authors read and approved the final manuscript.

Article Information

DOI: https://doi.org/10.9734/jabb/2024/v27i7969

Open Peer Review History:
This journal follows the Advanced Open Peer Review policy. Identity of the Reviewers, Editor(s) and additional Reviewers, peer review comments, different versions of the manuscript, comments of the editors, etc are available here: https://www.sdiarticle5.com/review-history/117231

Original Research Article

Received: 28/03/2024
Accepted: 01/06/2024
Published: 04/06/2024

ABSTRACT

Introduction: Removing nickel from wastewater is complex and costly, requiring research into alternatives with technical and economic solutions. An alternative would be the use of plant biomass. In the Northeast region of Brazil, one of these alternatives, with a large cultivation area, is

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Cite as: *Kramer, D. G., Sousa, A. M. de, & Júnior, G. B. C. (2024). Comparative Analysis of Nickel Adsorption by Natural Sisal Fiber and Treated with Citric Acid. Journal of Advances in Biology & Biotechnology, 27(7), 86–95. https://doi.org/10.9734/jabb/2024/v27i7969*

sisal (*Agave Sisaliana*), which may have greater yields for this purpose through surface chemical treatment with citric acid, which inserts carboxylic groups into the structure of the plant fiber. Thus, the objective was to analyze the ability to remove nickel in solution by sisal fibers.
Methodology: For this purpose, part of the sisal fibers was cut into 0.5 cm, washed and dried; initially treated with NaOH (0.1 mol/L); washed with distilled water and treated with citric acid (1.2 mol/L). The fibers were identified by FTIR; XRD; XRF and SEM. Adsorption tests were carried out with nickel solution for the parameters: kinetics, balance, influence of pH and adsorbent mass.
Results: The XRF analysis demonstrated a greater presence of NiO adhered to the treated fiber (55.74%); FTIR confirmed the presence of non-ionized carbonyl in the region from 1700 to 1750 cm-1; the adsorption kinetic test demonstrated greater effectiveness of the treated fiber > 80%; an ideal adsorbent mass was observed to be 0.1g; the ideal pH for removal was 5.0 while the equilibrium test has the best Languimuir isotherm, the uncontrolled fiber obtained R2 = 0.9810 and the controlled fiber R2 = 0.9643.
Conclusion: Therefore, it was observed that the treatment of sisal fiber with citric acid increases the removal efficiency of nickel ion in aqueous solution, presenting itself as a low-cost and widely available material.

Keywords: Sisal; removal; nickel; citric acid.

1. INTRODUCTION

The expansion of urbanization in developing countries has contributed to the contamination of water resources by heavy metals, such as nickel, chromium, copper, gallium, aluminum and cadmium, which are sources of pollutants: industrial sectors textile, tanneries, metallurgical, microelectronics, mining and electroplating [1,2]. These metals can pose risks to human health, including exposure to nickel, the object of study, allergic reactions (irritation of mucous membranes and skin), malaise (nausea, vomiting) and risks of carcinogenicity [3,4].

Removing this heavy metal from wastewater is complex and costly, requiring research into alternatives with technical and economic viability. An alternative would be the use of plant biomass, with great availability and the possibility of regeneration through adsorption [5,6,7,8].

In the Northeast region of Brazil, one of these alternatives, with a large cultivation area, is sisal (Agave Sisaliana). This plant, native to Mexico, was introduced to the country in the 20th century and is currently used for manufacturing ropes, automotive coatings and handicrafts [9,10,11]. Being chosen as a vegetable fiber for Ni (II) removal tests from natural and chemically treated aqueous solutions.

Surface modification is, in general, a resource that enables the evolution of a certain characteristic, leaving the material with greater affinity, consequently improving its performance [12,13]. There are several types of surface modification, ranging from bleaching, cationization, to the insertion of chemical groups. Among the forms of surface chemical treatment to improve the removal of metal ions is the use of citric acid, which inserts carboxylic groups into the structure of the plant fiber [6,14]. Thus, in an aqueous medium, the OH of the carboxylic group dissociates, forming negative charges, which will be binding points for metal ions in solution (Me+). Therefore, the efficiency of sisal fiber would be greater for removing Ni (II) from aqueous solutions after carrying out this chemical treatment [6,14,15].

Some studies point to efficiency in this process, including: functionalization of activated carbon to remove copper [15], modified agricultural residues to remove copper [12], modified coconut fiber for copper removal [6]. plant bark (*Ceiba pentandra*) for nickel removal [15] and sugarcane bagasse for lead adsorption [14]. The main objective of this work was to compare the nickel adsorption performance using sisal fiber modified with citric acid and unmodified sisal fiber.

2. MATERIALS AND METHODS

2.1 Fibers Collection and Preparation

The sisal fibers from Embrapa in Campina Grande – PB, Brazil, were first cut to a size similar to 0.5 cm, washed with non-ionic detergent for 60 minutes to remove impurities and undesirable dirt that accompanied the fiber during its collection. Subsequently, the fibers were properly dried in an oven (Forced

Convection Oven, BIOFOCO BF2 ECF 64) at a temperature of 115°C.

2.2 Chemical Treatment of Fibers

The sisal fibers were previously immersed in a sodium hydroxide solution (0.1 mol/L) with a fiber/solution ratio of 1g:40ml, where they were stirred for 2 hours. They were then washed with distilled water until the solution reached a pH between 6 – 7. Drying at 55°C for 24h was carried out before the next step.

The dried fibers were immersed in a citric acid solution (1.2 mol/L) in a proportion of 1g/20ml, stirred for 30 minutes and then dried for 24h at 55°C. To complete the treatment, the temperature was increased to 120°C for 90 min. A final drying at 55°C for 24h was carried out [5].

2.3 Biomass Characterization

Sisal fibers chemically treated and untreated sisal fibers were characterized by:

- FTIR - Spectroscopy in the Infrared Region (FTIR) to characterize the chemical and molecular nature of samples (resolution of 4 cm -1 and 32 scans) - NEXUS - 470 - FTIR;
- XRD – X-ray diffraction for analysis of the crystalline phases of samples (fast detector, type D, operating at 30 kV and 15mA, copper anode K α = 0.1542 nm and Nickel filter) XRD-7000 X-RAY DIFFRACTOMETER;
- FRX - X-ray fluorescence spectrometry that determines and quantifies which chemical elements are present in the fiber in percentage - EDX720 SHIMADZU ENERGY DISPERSIVE X-RAY SPECTROMETER;
- SEM – Scanning Electron Microscopy (Voltage 20-30 kV and magnification between 75 x and 30,000 x) - TM 3000 – TEBLETOP MICROSCOPE – HITACHE).

2.4 Adsorption Tests

To carry out the adsorption tests, solutions containing nickel and sisal fibers were used under stirring. In order to define the best processing conditions for adsorption, the following parameters were analyzed: kinetics, balance, influence of pH, where it was adjusted when necessary with solutions of sodium hydroxide (NaOH) and sulfuric acid, influence of mass of the adsorbent, where the time, rotation and volume of the solution remained constant, but with mass variation. The samples, after being collected, were filtered and stored under refrigeration for later analysis.

The essential calculations for the adsorption analysis were based on literature [1,4,16]. The metal removal capacity per gram of biosorbent (qe (mg/g) was calculated using the expression:

$$q_e = \frac{V\left(C_o - C_e\right)}{M} \quad (1)$$

Where C_0 and C_e are the initial and final concentration of the metal in solution (mg/L), V the volume of the solution (L) and M the adsorbent mass (g). The determination of the percentage removal rate (R%) of Ni (II) was calculated by the expression:

$$R\% = \frac{\left(C_o - C_e\right)}{C_o} \times 100 \quad (2)$$

Nickel concentration analyzes in aqueous solution, at all stages of the study, were carried out using flame atomic absorption spectroscopy (FASS) - Spectra equipment - Atomic Absorption Spectrometer 50 B.

a. Kinetics adsorption

This test was carried out to analyze the behavior of the adsorption process as a result of contact time. For this test, sisal fibers without chemical treatment and with chemical treatment were used. The pH was adjusted with NaOH (Sodium Hydroxide) or H2SO4 (Sulfuric Acid) when necessary to 5.0 using the MS TECNOPON pH meter.

In this experiment, 0.1g of fiber was stirred with 40ml of nickel solution at a concentration of 90mgL-1, in contact times ranging from 1 min to 24h (1 min, 2 min, 5 min, 10 min, 30 min, 60 min, 120 min, 240 min, 480 min, 960 min and 24h). The procedure was carried out in duplicate, on a CERTOMAT MO shaker, at a speed of 150 rpm. The resulting concentration after adsorption was determined.

b. Equilibrium adsorption

In this study, the influence of nickel concentration on its adsorption by sisal fibers with and without

chemical treatment with citric acid was evaluated. The tests were carried out using 40ml of nickel solutions at concentrations of 10 to 300 mg/L, with a pH equal to 5.0, adjusted when necessary with NaOH (Sodium Hydroxide) or H_2SO_4 (Sulfuric Acid), in which they were left under stirring. with sisal fibers for 4 hours, at a speed of 150 rpm.

c. Influence of pH

The influence of pH evaluates the ideal pH condition for greater metal adsorption. The tests were carried out in duplicate batches with 40 ml of 90mg/L nickel solution and pH varying from 2.0 to 6.0, adjusted with NaOH (Sodium Hydroxide) or H2SO4 (Sulfuric Acid), under agitation with the sisal fibers at a speed 150 rpm for 8h.

d. Influence of mass

This test was carried out to analyze the influence of the amount of sisal used in adsorption. 0.05g and 0.15g of adsorbent mass were placed in contact with 40ml of 90mgL-1 nickel solution and kept stirring for 6h. The procedure was performed in duplicate, at a speed of 150 rpm. After adsorption, the samples were filtered and the final concentration of the solutions was determined [1].

3. RESULTS AND DISCUSSION

3.1 Fiber Characterization

a. XRD

The comparative XRD analysis between the two fibers showed that the chemical treatment exposed the crystalline phase of the cellulose of the treated fiber, a fact that justifies a higher intensity profile for the treated fiber, illustrated in the Fig. 1. Mainly at the level of 22o, the highest intensity peak, typical of ligno-cellulosic materials [10,17].

b. XRF

The sisal fibers used in this study were evaluated by XRF after being exposed to a nickel solution. It is possible to observe that the metal is adhered to the surface of the fiber in large percentages, compared to other chemical elements, thus confirming the adsorption and retention capacity of the metal in solution [10,18]. As shown in Table 1, there was a higher concentration in chemically treated sisal fiber.

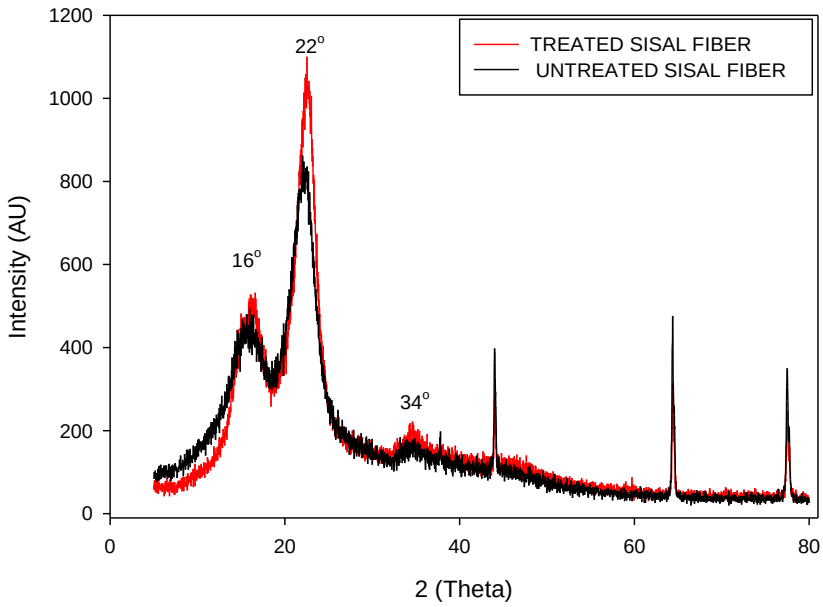


Fig. 1. X-ray diffraction analysis (XRD) of natural sisal fiber treated with citric acid
Source: Authors, 2024

Table 1. XRF analysis (untreated and chemically treated sisal) after the Ni (II) adsorption test in aqueous solution

Analyte	Untreated fiber	Treated fiber
Ni	47.233 %	55.740 %
CaO	27.974 %	31.591 %
Other components	24.793 %	12.669%

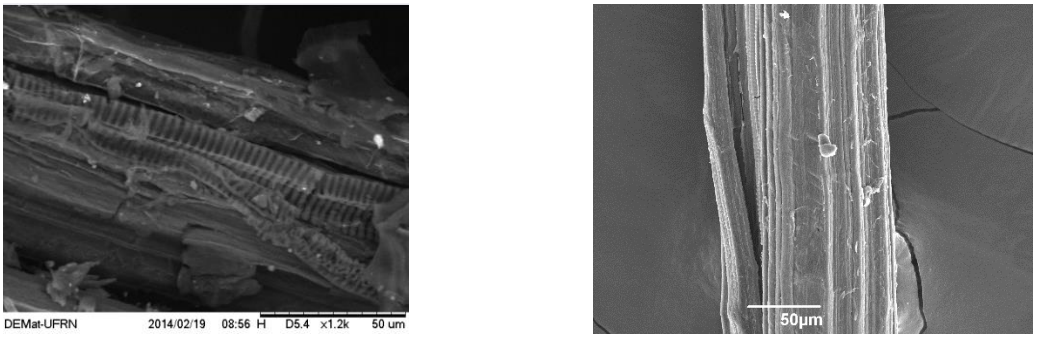
c. SEM

The superficial analysis of the sisal samples by electron microscopy (SEM) made it possible to confirm the effectiveness of the chemical treatment of the fibers, by removing impurities and undesirable dirt that accompanied the fiber during its collection. Chemical treatment with NaOH serves to remove impurities (particles and dust) and particles from the fiber surface.

Fig. 2 – B illustrates the surface of chemically treated sisal fiber free of impurities.

d. FTIR

Fig. 3 illustrates the FTIR analyzes of sisal samples, describing characteristic vibrations of chemical groups of these samples at: 3000-3500 cm -1 (hydroxyl groups - OH), 1010 and 1040 cm-1 (CO and CH), 1650 and 1750 cm-1 (carbonyl group). More specifically, the carbonyl belonging to a non-ionized COOH group has an absorption band in the region of 1700 to 1750 cm-1, with specific addition of these groups confirmed by FTIR [19,20].



(A) (B)

Fig. 2. SEM analysis of sisal fibers: Untreated fiber (A) and Chemically treated (B)

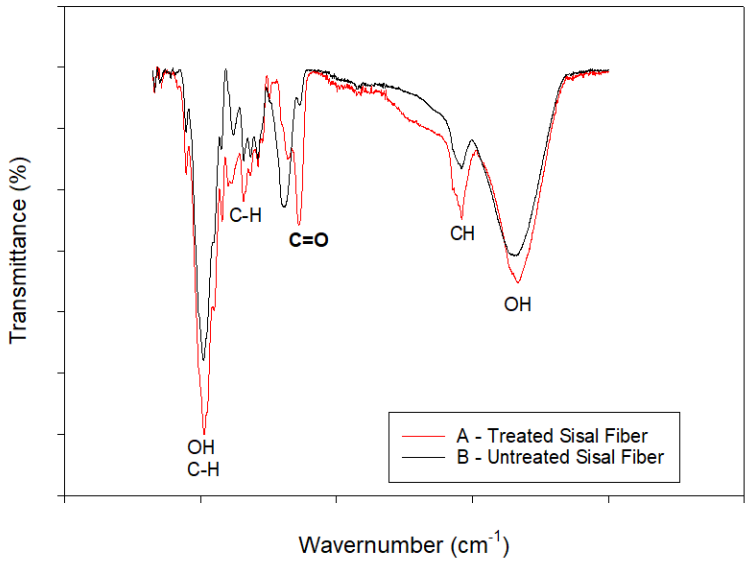


Fig. 3. FTIR analysis of sisal fibers: Untreated fiber (A) and chemically treated (B)

The chemical treatment of sisal fiber with citric acid followed by heating this biomass contributes to the condensation of citric acid anhydride in the vegetable structure. This added chemical has the COOH group, where the carbonyl C=O is located, which has an absorption band in the region of 1700 to 1750 cm^{-1} [5]. Thus, as illustrated in Fig. 3, the carbonyl in the treated fiber referring to the carboxylic acid group, when ionized, presents electrostatic reactions with metal ions in aqueous solution, favoring their removal [5].

3.2 Adsorption Tests

a. Kinetics

The kinetic study aimed to evaluate the progress of the adsorption process until reaching the concentration limit of Nickel ions adsorbed on the fiber surface under the influence of time – Fig. 4. The adsorption of Ni under the experimental conditions of this study allowed us to observe that the process occurs in less than 30 minutes. It was possible to verify that the ability to remove this metal occurs quickly in both sisal samples, being, however, more efficient for the treated sisal fiber. Thus demonstrating that the insertion of carboxyl groups would increase the reactive surface area of this sample for capturing the nickel ion in solution.

In the literature, different biomasses (coconut fiber [21]; vegetable biochar [22]; modified soy hulls [23] and sugarcane bagasse [24]) are reported for nickel absorption in aqueous solution, with percentage removal rates above 80% and equilibrium reached before 10 minutes of contact, similar to the present study, proving to be a cheap and efficient option for this purpose.

b. Influence of mass

The investigation of the effect of the amount of biomass on the removal of metal ions was carried out using 0.05, 0.1 and 0.15g, in contact with 40ml of a solution containing 90mg/L of nickel for 6 hours. It is possible to determine an ideal sample in 0.1g – Fig. 5.

According to the illustration above, it can be seen that the main adsorption range is 0.1g. The justification for the lower adsorption in ranges below 0.1g of biomass would be the few options for adsorption sites, affecting its efficiency. However, for larger amounts of biomass there is no significant variation. This can be explained by the fact that active sites overlap with a higher amount of biomass [1].

c. Influence of pH

The pH of the solution is an important parameter to be analyzed as it directly influences the adsorption efficiency. To verify an optimal pH for adsorption of metal ions, a study was carried out with different pH values, shown in Fig. 6.

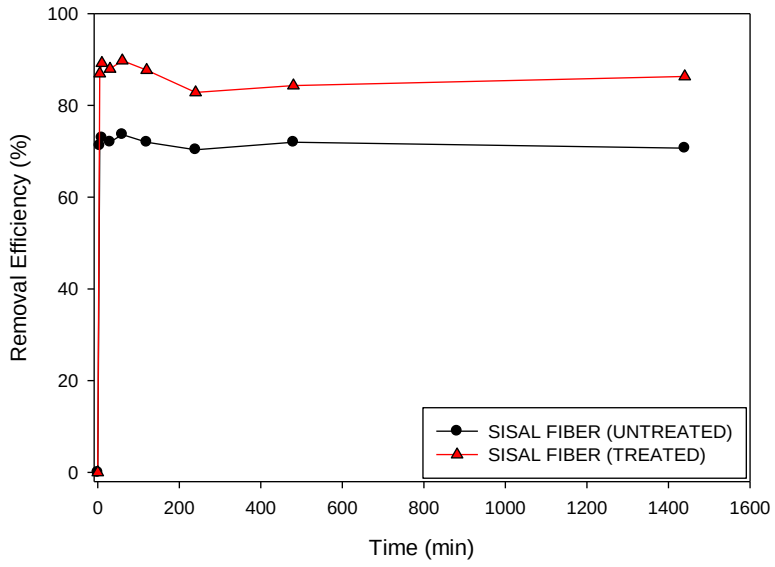


Fig. 4. Effect of time on Ni (II) removal by chemically treated and untreated sisal fiber

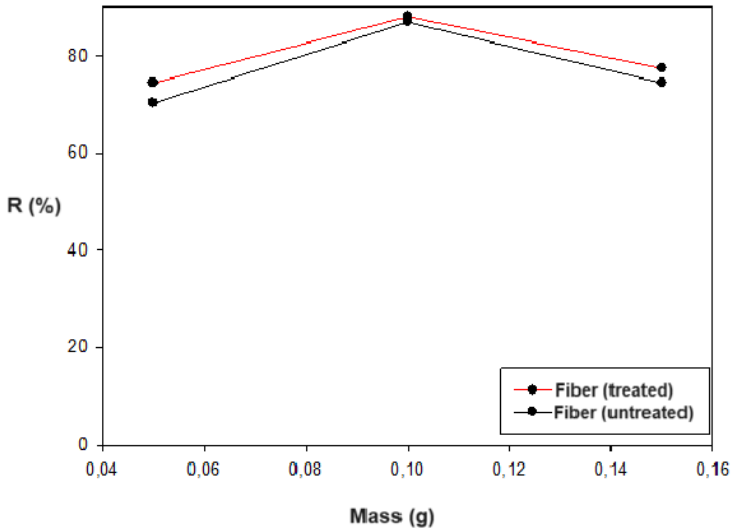


Fig. 5. Influence of the adsorbent mass (sisal) on the adsorption of nickel (II) in solution. R% (Removal efficiency)

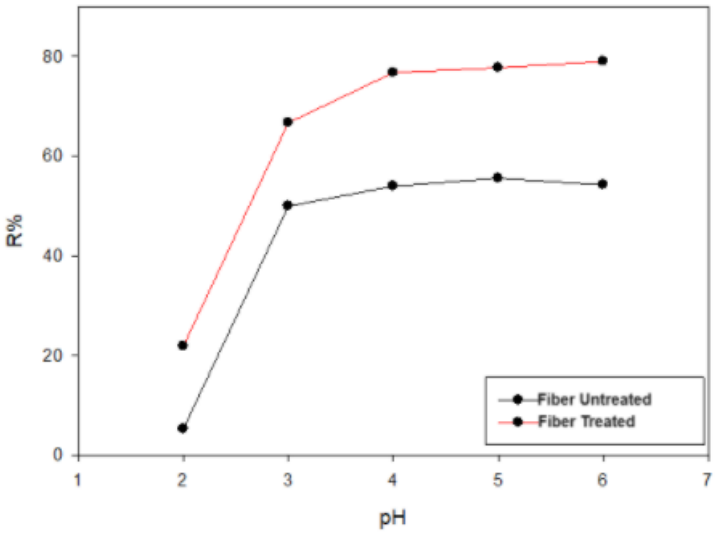


Fig. 6. Influencia do pH na adsorção do níquel por fibras de sisal

The chemically treated fiber obtained better results in adsorption of nickel ion in solution. At this stage, it is observed that for very acidic pH values, such as the range between 2.0 and 4.0, there is little efficiency in the adsorption of metal ions. It is possible to find in the literature that low pH values correspond to a higher concentration of free H^+ ions in the solution. As the pH increases, this concentration decreases. In the ion exchange process, the more acidic the solution, the greater the competition for H^+ ions and metals to be adsorbed, making their adsorption more difficult [6,14,24].

d. Equilíbrium

In the equilibrium study and obtaining adsorption isotherms, experiments were carried out varying the Ni (II) concentration (10 to 180 mg/L). The models evaluated were those of Langmuir and Freundlich [7,16]. The Langmuir model was determined using the equation:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (3)$$

Where qe is the equilibrium sorbate absorption (mg/g), qm is the maximum binding capacity

(mg/g), Ce is the equilibrium concentration of sorbate in solution (mg/L) and KL is the equilibrium constant apparent (L/mg).

The Freundlich isothermal model can be written as indicated in Eq.(4):

$$q_e = K_f C_e^{1/n} \quad (4)$$

Where qe is the amount of adsorbed metal (mg/g), Ce is the equilibrium concentration in the solution (mg/L), kf is a constant related to the adsorption capacity (mg/g) and 1/n the adsorption intensity.

Fig. 7 illustrates the Languimuir and Freundlich isotherm for a chemically untreated sisal sample. The second model (R2 = 0.9810) being considered a better correlation with the experimental and calculated data, the Languimuir model. The other parameters of this model were calculated at Kpf = 3.1268 and n = 0.5477.

Fig. 8 illustrates the Languimuir and Freundlich isotherm for a chemically treated sisal sample. The first model (R2 = 0.9643) of Languimuir is considered a better correlation with the experimental and calculated data. The other parameters of this model were calculated at K= 0.0864 and qmax = 64.58.

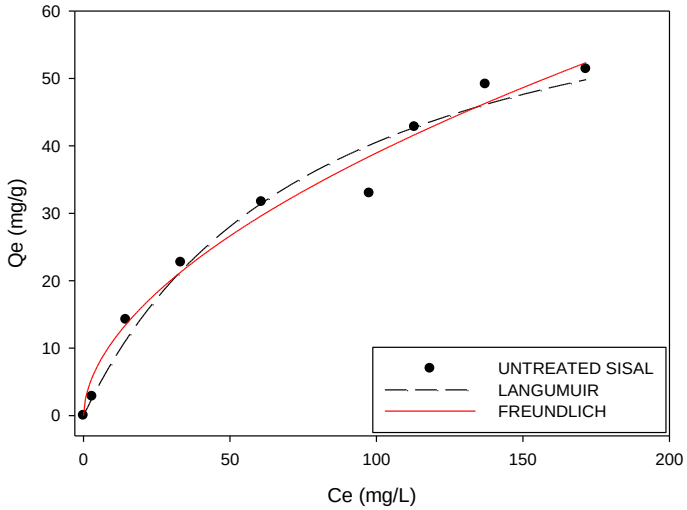


Fig. 7. Analysis of the Ni(II) adsorption isotherm by chemically untreated sisal fiber

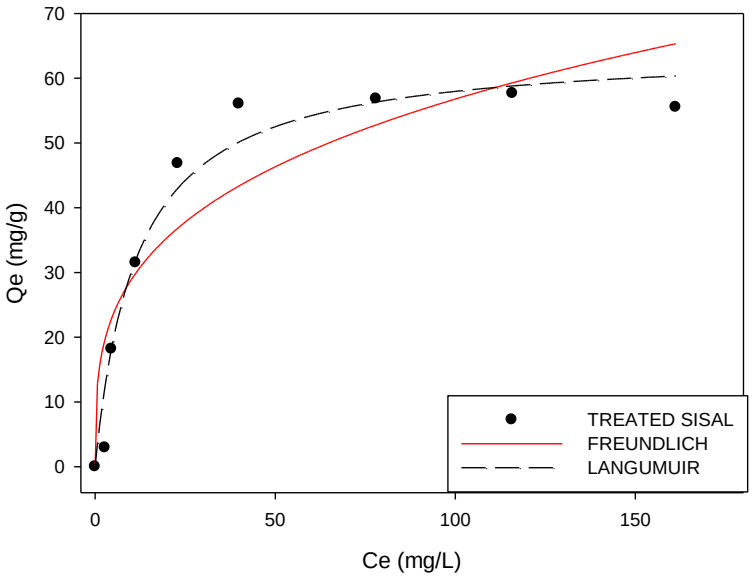


Fig. 8. Analysis of the Ni(II) adsorption isotherm by chemically treated sisal fiber

The Freundlich model is valid for multilayer adsorption, with mutual interactions between sorbed metal cations and biomass. In this case, it is expected that adsorption will be greater due to the increase in the concentration of the metal ion in the solution, although in laboratory practice, a saturation limit is observed in this process. The Langmuir model assumes the occurrence of specific active sites in the biosorbent for the binding of metal cations in solution, with the type of electrostatic interaction being in a monolayer [22,25,26,27]

As the results of the present study are better correlated with the Languimuir model, the type of homogeneous adoption is explained, where Ni^{+2} ions bind to the active sites of the applied biomass [22,25,26,27]. At Isotherma, a higher initial nickel concentration of 300 mg/l was observed for residual nickel in solution for untreated sisal at 171.55 mg/l, while for treated biomass it was observed at 161.20 mg/l.

The data from this research are similar to the findings of Shen et al. [22], who produced bicoal using rice husks to adsorb nickel, with better adsorption being considered at pH above 4.0; reaching equilibrium in less than 5 minutes and adsorption percentage above 80%. Furthermore, a similarity was observed with the study by Praveen et al. [26] who applied brick bioassay for nickel remelting in aqueous solution, observing a rate greater than 80%; equilibrium time less than 10 minutes.

Nickel in wastewater is a toxic element and can lead to carcinogenicity, [2] and the efficiency of using biomass (sisal fiber) was demonstrated in the present study with removal rates above 80%. In kinetics, the final concentration of Ni(e) in solution for treated sisal biomass was observed at 12.32 mg/L and untreated sisal at 26.40 mg/L. This, with low-cost chemical treatment, abundant and cheap biomass and easy handling, as a potential use in the treatment of effluents containing nickel (II) ions.

4. CONCLUSION

The data presented in the study made it possible to confirm through SEM, XRD and FTIR that the chemical treatment of sisal fiber was efficient, demonstrating greater crystallinity, absence of dirt and the presence of the carbonyl group. These aspects contributed to greater efficiency in the removal of Nickel (II) in aqueous solution by the chemically treated fiber, presenting a higher percentage in the XRF test and better performance in the kinetic tests and influence of pH.

Therefore, sisal fiber presents itself as an efficient, low-cost and highly available biomass for application in the removal of Nickel in aqueous solution.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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